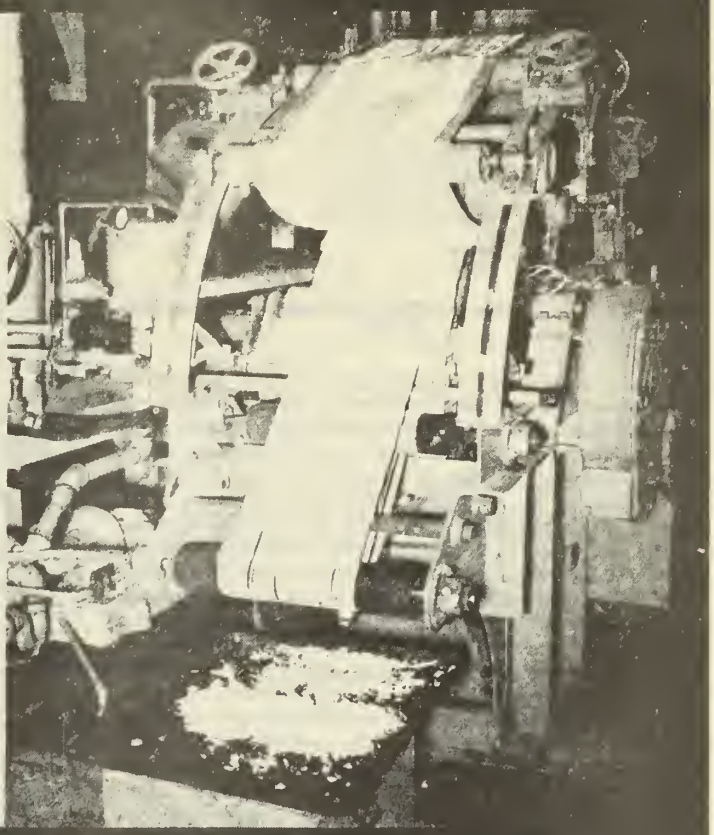
SOYBEAN PROTEIN PRODUCTION

Comparison of the Use of Alcohol-Extracted with Petroleum-Ether-Extracted Flakes in a Pilot Plant[1/]

The pilot-plant production of soybean protein is reported for the first time. It is shown that a process consisting of extraction of soybean flakes, separation of suspended solids from the dispersion, precipitation, and dewatering may be pursued in a straightforward manner with satisfactory results. The fractions into which the soybean flakes are separated during the processing, as well as their sizes, are given. A comparison is made of the production of protein from flakes resulting from the extraction of the oil with petroleum ether in one case and with alcohol in the other. The superiority of ethanol-extracted flakes for processing soybean protein is noted.~~The photograph shows discharge of protein from vacuum-drum filter



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THE pilot-plant production of soybean protein is relatively simple as is its laboratory preparation; but the steps which lend simplicity to the laboratory process cannot be directly changed to large-scale production methods. The laboratory process for preparing protein consists essentially of extraction of solvent-extracted soybean flakes with water or dilute sodium hydroxide, removal of the undispersed part by centrifugation, and precipitation of the protein with acid at a pH between 4 and 5. Detailed descriptions of this procedure and the properties of the protein were described previously (*6*, *7*). In transferring to pilot-plant production, certain characteristics of the materials become increasingly important. For example, the crude soybean meal mixture contains a mucilaginous substance which defies all attempts at filtration; the drying of large quantities of protein emphasizes its heat sensitivity as evidenced by heat denaturation (*1*); and under some conditions, foaming is a serious problem. Factors governing these and related problems are presented in this paper.

This labor tory has been operating a pilot plant (Figure 1) for more than a year, and its description will serve as a guide to large-scale production of soybean protein. The process consists of four steps: extraction of protein, separation of dispersion from suspended solids, precipitation, and dewatering.

Soybean flakes and the extracting medium, consisting of water or dilute alkali as desired, are stirred together in jacketed tank 1 for the time and at the temperature selected (*5*, *7*). The dispersion is separated from the coarse particles of the flakes on gyrating screen 2. The dispersion is pumped to storage tank 3, and the residual flakes are pressed and dried. The dispersion from storage tank 3 is usually passed through centrifuge 4 for clarification, and then pumped to precipitation tank 5 where acid is added to bring the pH to the desired point for precipitation of the protein. The precipitated protein is allowed to settle to the bottom of the tank, and the supernatant whey is siphoned off. To wash the protein, water is added and the mixture stirred and allowed to settle again after the pH is adjusted to the original precipitation value. The siphoning and washing may be repeated, or the thick slurry remaining after siphoning of the wash water may be piped to string filter 6. The resultant protein cake from the filter is ready for drying.

RAW MATERIALS AND EXTRACTION

The best raw material for the manufacture of soybean protein is probably the residue resulting from the solvent extraction of flaked soybeans. These flakes are used in preference to expeller meal because they have a lower oil content and contain a more soluble and lighter-colored protein. The greater solubility and lighter color are results of lower and more uniform temperature

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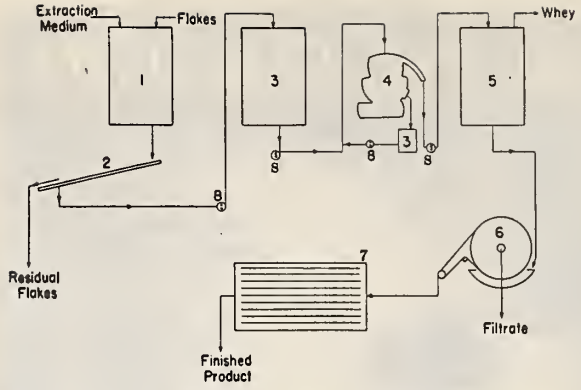

Figure 1. Flow Diagram for Pilot-Plant Production of Soybean Protein

treatment in the solvent-extraction process. The flakes available commercially have been extracted by petroleum ether. It has been known for some time (*2*) that the conditions of oil removal have a considerable influence on the character of the protein obtained. In addition, we have found that the method of oil extraction has a profound effect on the properties of the protein, greatly influences the ease and speed of the protein manufacture, and consequently determines its economics. During a study of the effects of the method of oil extraction of the color, odor, and taste of soybean meal, ethanol was found far superior to all other solvents studied (including the petroleum ethers) when used on flakes having a comparatively high moisture content. Furthermore, the production of protein from the ethanol-extracted flakes was considerably speeded. For this reason the pilot plant study was made on both the ethanol-extracted and the petroleum-ether-extracted flakes. The protein content of the ethanol-extracted flakes is about 50%, and that of the petroleum-ether-extracted flakes about 45% on the air-dry basis. Since soluble calcium and magnesium salts have a particularly bad effect (*7*) on the dispersibility of the protein, the other principal raw material, water, should have as low a soluble salt content as possible.

Extraction conditions are described in other publications (*5*, *7*). All extractions in this study were made at a water to flake ratio of 20 to 1 by weight and at a temperature of 30° C. with an extraction time of 30 minutes after reaching the specified temperature. The size of the batch of flakes varied from 60 to 100 pounds.

SEPARATION OF DISPERSION FROM FLAKES

In view of the apparent impossibility of filtering the flakes from the extraction medium by any method which would permit the residue to be processed for animal feed, the only recourse seemed to be the use of some type of centrifuge or of a screening operation. The moisture in the residual flakes from the screening operations can be reduced slightly by pressing at a few hundred pounds per square inch, but a considerably higher pressure applied for a long time, has only a slight additional effect. In view of this fact, and the large quantity of material to be handled, it would seem that a screening operation followed by continuous pressing constitutes a more desirable procedure than the use of centrifugal equipment.

In the pilot plant a gyrating screen, 2 × 4 feet, tilted at an angle of 6°, is used to remove coarse solids from the protein extract. Two baffles, each 2.5 inches wide, cross the screen at about one fourth and one half the distance from the upper end and serve to spread the slurry over the entire screen. The factors affecting the performance of the screen are: pH of slurry, size of screen openings, rate of feeding slurry, and means of extracting flakes before their use in making slurry. The measurement of the performance of the screen is the quantity of suspended solids passing through. Of equal interest are the volume and concentration of the extract passing over the screen. A limiting factor in the rate of screening is the formation of foam, which can be controlled by limiting the rate of feed of the slurry or by adding antifoaming agents. In the studies reported here, antifoaming agents were not used.

The variations in results attributable to a difference in the mesh of the screen are given in Table I. The feed rates indicate the maximum rate for continued operation in our apparatus without foaming. As would be expected, the feed rate increases with a decrease in mesh number, but the quantity of suspended solids passing through the screen also increases. If there were no further clarification, the protein would be contaminated to the extent indicated in line 6 (percentage of suspended solids on the protein). For certain purposes a protein of this quality might be satisfactory. When further centrifugal clarification is used, the increase in recoverable dispersion with decrease of mesh number (line 7) and the feed rates (line 1) become appreciable factors, since the suspended solids indicated in line 3 are in no case excessive. The differences in suspended solids would affect only the time during which a bowl-type centrifuge could be operated without shutting down for cleaning. For a given centrifuge and a given dispersion, the several times of operation would be proportional to the weight values of line 3. These suspended solids, after recovery by centrifuging, may be combined with the wet mash passing over the screen when the wet mesh is to be dried and used as stock feed.

Table I. Performance of Gyrating Screen with Petroleum Ether Flakes Extracted at pH 9.5

Screen Size in Standard Mesh	40		80		150	
1. Feed rate, gal. per hour	277		159		128	
Materials Passing through Screen	%[a]	Wt.[b]	%[a]	Wt.[b]	%[a]	Wt.[b]
2. Total solids	3.74	0.67	3.68	0.65	3.45	0.53
3. Suspended solids	0.45	0.089	0.32	0.056	0.29	0.049
4. Dissolved solids	3.29	0.59	3.36	0.59	3.16	0.54
5. Precipitable protein	1.80	0.322	1.74	0.306	1.74	0.316
6. Suspended solids on protein	25.0	...	18.4	...	16.7	...
7. Dispersion recovered	66.4	...	87.5	...	86.8	...
8. Whey solids	...	0.26	...	0.29	...	0.22
Materials Passing Over Screen						
9. Water	89.4	...	89.7	...	89.8	...
10. Total dissolved protein[c]	11.6	...	12.5	...	13.7	...
11. Recoverable dry residus	...	0.33	...	0.35	...	0.41

[a] Percentage of dispersion.
[b] Weight per unit weight of original flakes.
[c] Total dissolved protein passing over screen, as percentage of total dissolved protein.

The effect of hydrogen ion concentration on the separation of the mash from the dispersion is given in Table II. The extractions at pH 6.6 were made with distilled water; those at 7.3 pH, with 0.3% sodium sulfite; those at pH 9.8 and 9.5, with 0.1% sodium hydroxide; and those at pH 10.4, with 0.2% sodium hydroxide.

With increasing pH there is an increase in the weight of precipitable protein per unit weight of flakes (line 11); there is also an increase in soluble solids not recoverable as protein (line 12 and suspended solids passing through the screen (line 9). There is a corresponding decrease in the solids passing over the screen (line 7). With increasing pH of extraction (line 5), the moisture in the mash increases slightly and the removal of liquid by pressing becomes more difficult. When the mash is to be used as stock feed, it would seem advisable to neutralize the alkalinity; this would reduce the difficulty of pressing at the same time. Under a gentle pressure of 2 pounds per square inch for 5 minutes,

a water-extracted mash having an original water content of about 88% was reduced to 81.3%; in another determination, a pressure of 224 pounds per square inch for 10 minutes reduced the water content to 70%; a pressure of 336 pounds per square inch for 90 minutes reduced the water content to about 60%. The protein content of the mash on the dry basis is given in line 15.

CLARIFICATION

The dispersion is clarified at 7600 r.p.m. by a disk centrifuge of the valve-bowl-concentrator type, with automatic control of the solids discharge. The sludge discharge is continually recirculated; this operation reduces the dispersion loss to a small quantity.

An idea of the contamination of the protein due to unremoved suspended solids may be obtained by comparing lines 6 and 14 of Table II. The former gives the contamination when the protein is separated without clarification in the centrifuge, and the latter, after clarification. At the lower hydrogen-ion concentrations the clarification is satisfactory; at the higher pH values it is not. The clarification indicated by comparing lines 3 and 13 is excellent in all cases; only because the ratio of liquid to protein is large does the small quantity of suspended matter rise to 8% when calculated as an impurity based on the quantity of protein (last figure in line 14).

The clarification for each extraction was studied at three feed rates—about 200, 300, and 500 gallons per hour. The removal of suspended solids was slightly less efficient at the higher rates, in which cases the limiting factor was not the clarification but the formation of foam in the centrifuge. A satisfactory rate for our equipment was between 275 and 300 gallons per hour, and the data in lines 13 and 14 of Table II were obtained at this feed rate.

Table II. Effect of Hydrogen-Ion Concentration on Wet Screening of Soybean Flake

(Screen size, 80 mesh; water-flake ratio, 20–1; temperature, 30° C.; time of extraction, 30 minutes after reaching temperature)

Flakes Extracted with:	Alc.[a]	P.E.[b]	Alc.	P.E.	Alc.	P.E.	Alc.	P.E.
Feed rate (gal./hr.)[c]	720	80	425	225	450	160	500	130
Before Clarification								
1. pH of protein extraction	6.6	6.6	7.3	7.3	9.8	9.5	10.4	10.4
2. % of dispersion as total solids	2.79	3.21	2.61	3.63	3.52	3.68	3.91	4.16
3. % of dispersion as suspended solids	0.26	0.24	0.30	0.29	0.31	0.29	0.49	0.36
4. % of dispersion as dissolved solids	2.53	2.97	2.31	3.34	3.21	3.36	3.42	3.80
5. % of moisture in mash	88.8	88.6	88.3	89.4	90.0	89.7	90.8	90.4
6. % suspended solids on protein	16.5	14.6	19.0	16.5	17.5	16.7	....	19.4
Weight per Unit Weight of Starting Flakes								
7. Of solids over screen	0.55	0.45	0.58	0.36	0.40	0.35	0.31	0.22
8. Of total solids through screen	0.449	0.547	0.418	0.624	0.599	0.648	0.689	0.77
9. Of suspended solids through screen	0.042	0.041	0.048	0.050	0.053	0.056	0.059	0.050
10. Of dissolved solids through screen	0.403	0.506	0.370	0.575	0.546	0.591	0.603	0.710
11. Of precipitable protein through screen	0.252	0.280	0.253	0.301	0.301	0.306	0.318	0.348
12. Of soluble solids (not recovered as protein)	0.156	0.226	0.117	0.274	0.248	0.528	0.285	0.281
After Clarification (About 300 Gal. per Hour)								
13. % of dispersion as suspended solids	0.001	0.001	0.001	0.001	0.01	0.004	0.08	0.15
14. % suspended solids on protein	0.06	0.06	0.06	0.06	0.56	0.23	2.28	8.08
Mash over Screen								
15. % protein (dry basis)	51.8	43.8	49.8	36.3	44.4	34.4	44.7	32.7

[a] Ethanol.
[b] Petroleum ether.
[c] For rates without foaming difficulties.

PRECIPITATION, SETTLING, AND WASHING

The precipitation of the protein is usually performed by adjusting the pH to about 4.5. Since the precipitated protein probably consists of several individual proteins having slightly different isoelectric points, it is probable that a change in either direction in the pH of precipitation would cause a change in the character of the precipitated protein.

The reasons for adopting the elementary process of settling are the necessity for removing an appreciable amount of soluble material from the dilute suspension, and the difficulty of filtering the precipitated protein.

The percentage of precipitable protein in the dispersions varied from 1.57 to 1.86%, and the percentage of other soluble solids (*2*) varied from 1.0 to 2.0%. The procedure of settling, decanting the whey, and washing by decantation is a necessary preliminary to filtration, since the low concentration of protein in the suspension is not readily filtered, and the nature of the cake is such as to make washing on the filter difficult or impracticable.

The time required for settling and the settling capacity govern the size of the other units, which can operate more or less continuously.

When the percentage of solids in the settled curd (uniformly mixed) is plotted against the time of settling, the slope of the curve gives the rate of settling. These curves (Figure 2) contrast the behavior of a water-extracted protein (pH 6.6) from ethanol-extracted flakes (curve 1) with those of proteins extracted at pH 6.6, 7.4, 10.6, and 11.4 (curves 2, 3, 4, 5) from petroleum-ether-extracted flakes, and with that of a protein extracted at pH 11.4 from ethanol-extracted flakes. The quantity of sol-

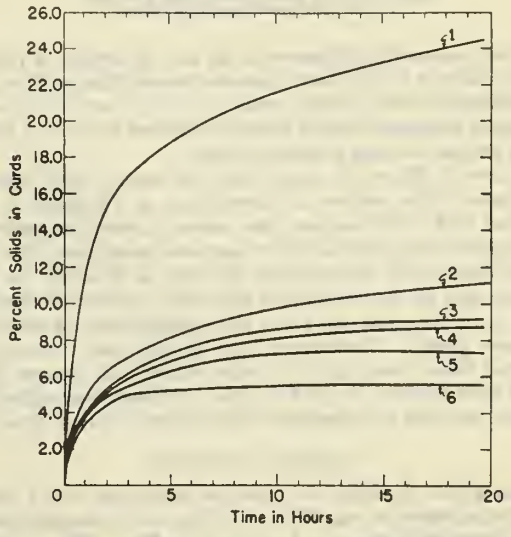

Figure 2. Settling Rate of Precipitated Protein

Curves 1 and 6 from ethanol-extracted flakes at pH 6.6 and 11.4, respectively; curves 2, 3, 4, and 5 from petroleum-ether-extracted flakes at pH 6.6, 7.4, 10.6, and 11.4, respectively.

uble solids, other than protein, increases with increasing pH (line 12, Table II), and so may change the viscosity and density of the medium through which the protein settles. Since a considerable portion of the readily soluble carbohydrates have been removed from flakes from which oil was extracted with ethanol there is an appreciable difference in the quantities of soluble solids extracted at the lower hydrogen-ion concentrations from the two kinds of flakes. The influence of increasing pH is greater on the ethanol-extracted than on the petroleum-ether-extracted flakes; this effect is shown in line 12 of Table II and in Figure 2, where curve 6 for protein extracted at pH 11.4 from ethanol-extracted flakes is the lowest of all.

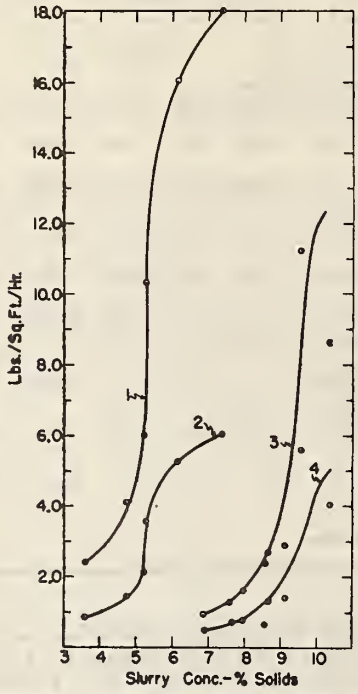

Figure 3. Filtration Rate of Protein

Curves 1 and 2 on basis of wet and dry protein from ethanol-extracted flakes; curves 3 and 4 for protein from petroleum-ether-extracted flakes.

The considerable difference in the rate of settling of protein extracted at a low pH from ethanol-extracted flakes and the rate of settling of other proteins, seems to result in part from a more granular structure which is known to increase the ease of filtration but has not been evaluated further.

Curve 1 (Figure 2) shows that the protein from ethanol-extracted flakes reaches a concentration of 6% solids in the settled curd in 20 minutes; the nearest approaching curve, 2, for protein from petroleum-ether-extracted flakes shows that the curd attains this concentration of solids in 2 hours. In the former case the concentration of 6% solids is reached six times as fast as in the latter, and above this concentration the factor increases greatly. Furthermore, the protein from ethanol-extracted flakes compacts to a concentration much greater than that ever attained by protein from petroleum-ether-extracted flakes, and this fact translates into economies during filtration.

FILTRATION AND DRYING

The protein is filtered by a vacuum drum filter with a string discharge (shown on page 799). The filter may be operated either as a string discharge or as a scraper-type filter. There is little difference in the operational effectiveness of the two, except that the string discharge presents a thin layer of curd in excellent condition for drying, whereas the scraper causes the curd to bunch up into masses which caseharden in the drying operation.

Significant data on the filter used for these experiments are: Diameter of drum, 3 feet; width of face of filtering surface, 1 foot; filtering area, 9.43 square feet; drum speed, 0.375 r.p.m.; proportion of drum immersed in the slurry, 1/4; string separation, 1/4 inch; and cake thickness at maximum filtration rate, about 5/16 inch.

The rate of filtration, given as pounds of protein per square foot of filtering surface per hour for a range of slurry concentrations in percentage of solids, is presented in Figure 3. Curve 1 shows the variation in rate of filtration with slurry concentration for a protein from ethanol-extracted flakes, the protein being in the wet condition at the time of discharge. Curve 2 represents the same yield translated to the dry condition. Curves 3 and 4 give the corresponding data on protein from flakes from which the oil had been extracted with petroleum ether. The difference in the rate of filtration, as well as the lower slurry concentration at which filtration is possible, is probably a reflection of the more granular structure of the protein from alcohol-extracted flakes.

The water content of the protein curd discharged by the filter varies from 60 to 80%, depending on the nature of the protein, the thickness of the slurry, the depth of slurry in the filter sump, and the rate of rotation of the drum. Under normal conditions the water content will be about 70% of the curd.

The drying of the protein is carried out in a conventional tray dryer with the temperature maintained at 120° F. The granular type of precipitated protein dries rapidly and uniformly, especially when in the condition resulting from the string discharge on the filter. The effect of heat and moisture on the denaturation of the protein in soybean meal has been studied (*1*), but the specific effects of various methods of drying the isolated protein are still under investigation.

DISCUSSION

The described processing of solvent-extracted soybean flakes for protein was on a scale large enough to indicate the behavior to be expected in large-scale production. In the case of extractions with water or at a low pH we would expect that (*a*) about one fourth of the weight of flakes processed would appear as dry protein; (*b*) about half of the flakes would reappear as dried, extracted mash with a small additional amount (about one twentieth) from the centrifugable solids; and (*c*) about one sixth to one fifth of the starting material would exist in the whey which is siphoned from the protein after the settling operation as soluble carbohydrates, proteoses, albumins, and other soluble substances.

The relatively large proportion of the flakes that reappear as dried, extracted mash could go back into stock feed channels at the price of the starting material and thus reduce by a corresponding figure the cost of the original flakes. However, this mash has approximately the same protein content as the original flakes because, during the extraction of a portion of the protein, a corresponding proportion of the other soluble solids was removed. The removal of a large proportion of the water-soluble constituents from the mash yields a product that is useful in plastics and adhesives; this type of material has been used in the soybean plastics developed by this laboratory (*3*, *4*). In any case, the higher protein content should command a premium over the original price. The whey solids have not been investigated to any extent.

The uses for which the protein is intended and the trade channels through which it moves determine, to a considerable extent, the details of the process used in its manufacture. For example, the protein mentioned earlier, which could be prepared by eliminating the clarification operation, can be prepared cheaply and can be used in the adhesive trade, as well as in certain paper coatings. For a superior product the clarification operation is essential, and for the best color, as far as we know now, the

protein must be prepared from flakes from which the oil has been extracted with alcohol. The color of products prepared from this latter type of protein is, for all practical purposes, equivalent to those prepared from casein, and the yellowish tinge is no longer a problem.

Any cost estimate would be premature until certain other factors are known. Among these factors are yield of protein under optimum conditions, effects of water-flake ratio and temperature on the processing of the protein, and optimum conditions for drying both the protein and the mash. These problems are now being studied in this laboratory.

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